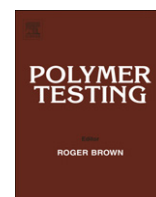


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Sample preparation

Tooling for near net-shape compression moulding of polymer specimens

Davide S.A. De Focatiis*

Division of Materials, Mechanics and Structures, University of Nottingham, Nottingham NG7 2RD, UK

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ABSTRACT

This study presents several designs for flash moulds intended to aid the damage-free production of polymeric specimens for mechanical and structural characterisation. The designs consist of interlocking metal parts that produce appropriately shaped cavities in which polymer specimens are moulded, and that are easily dismantled after moulding to allow removal of the specimens from the moulds. Very limited sample preparation is required after removal of the specimens. Several of the proposed designs have been manufactured and successfully employed in the production of rectangular specimens for characterisation of a range of polymers.

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1. Introduction

The experimental material scientist has an intrinsic need to be able to produce and reproduce test specimens with well defined geometries in order to measure the structural and mechanical response of materials. The reason for this is that a large number of material properties are typically obtained from functions of both measured experimental quantities such as forces and displacements and specimen dimensions such as lengths, widths and thicknesses. Therefore, the accuracy of the determination of such material properties is often dependent on precise measurements of the geometry of the test specimen. The experimentalist is responsible for the generation of the experimental test data necessary to aid the development of new theories and for the validation of numerical models. He or she is also responsible for providing the practicing engineer with the precise material parameters required by such models so that *virtual* testing of parts in service can be carried out in shorter times and at a lower cost than the equivalent additional experimental tests. In short, the experimentalist plays several important roles within engineering and materials design, and producing high quality test specimens for characterisation forms a critical

part of realising the reproducible and high-quality experimental data desired by all concerned.

One important and widely used laboratory technique for the manufacture of solid polymeric specimens is compression moulding. This is the process of heating a given polymeric material to above its glass transition (for amorphous polymers) or above the crystal melting point (for semi-crystalline polymers), under moderate pressure for a prescribed time, in a shaped cavity and, subsequently, cooling the material to induce solidification in the new shape. The polymer fills the shaped cavity, known as a mould, whilst in the molten state, and must be removed from the mould after it has solidified, often for further shaping prior to testing. The process of removal of the moulded polymer specimen from the typical metallic mould is not always straightforward because of adhesion between polymer and metal, and because of the constraints imposed by the shape of the metal mould parts. Adhesion between polymer and metal mould is a more significant issue with oxygen-containing polymers [1]. If significant stress has to be applied to the moulded polymer during the removal, it is possible that this will induce viscoelastic or even plastic deformation, and in extreme cases even crazing and cracking, invalidating subsequent mechanical testing. If separate post-moulding machining is required for the production of polymeric test specimens from moulded sheets or blocks, one needs to contend with the friction

* Tel.: +44 115 9514097; fax: + 44 115 9514115.

E-mail address: davide.defocatiis@nottingham.ac.uk.

arising from the cutting tools, and the consequent change in temperature of the polymer, which can alter the structure and thermal history of the specimen being investigated. Although this can be erased by appropriate subsequent annealing, it is time-consuming, and several cycles of machining and annealing may be required due to the release of residual stresses. Additionally, machining of specimens from sheet material generates waste polymer, which can be of concern if the material is particularly precious, or if the original synthesis was lengthy and complex.

In this paper, we propose a series of designs for the manufacture of tooling suitable for near net-shape compression moulding that specifically addresses all of the above issues by virtually eliminating the need for post-moulding specimen preparation prior to testing. The introduction describes the process of compression-moulding, including mould design and the important thermal transitions of the polymers involved. The design of a range of novel moulds for near net-shape specimen production is then presented. We then report on our own experience in manufacturing and utilising the novel mould designs for the production of specimens from a range of polymeric materials, and discuss the implications of these observations. Finally, we present a series of conclusions and recommendations.

2. Background

2.1. Compression moulding

Compression moulding is one of the simplest methods of manufacture of polymer specimens, and widely used in laboratories worldwide. It is a batch production technique whereby polymeric material is heated within a cavity, typically but not always metallic, under light to moderate pressure applied by a mechanical or hydraulic press. The required mass of polymeric material to be moulded, typically in the form of granules, powder or solid fragments, is placed within the cavity of the mould. Heat is then applied to the platens and, by conduction, to the mould and polymer. The polymeric material spreads to fill the cavity because of the flow of the polymer resulting from the applied pressure. After a suitable time, the mould containing the polymer is cooled, either naturally or through forced convection of air or water in suitably drilled channels in the press platens. The mould is then opened and the process of carefully removing the specimen from the mould can begin. Fig. 1 illustrates the typical sequence of events that takes place during compression-moulding.

Compression moulding is one of a range of production methods suitable for polymeric specimens that include injection-moulding and extrusion. It differs from other methods in that only limited shearing and mixing of the polymer melt occurs. In general, this is precisely the aim of compression moulding – to produce homogeneous, isotropic test specimens, that cannot be reliably produced using other techniques [2].

2.2. Mould designs

There are two broad classes of mould design: flash (or picture-frame) moulds, and positive moulds [2]. Flash

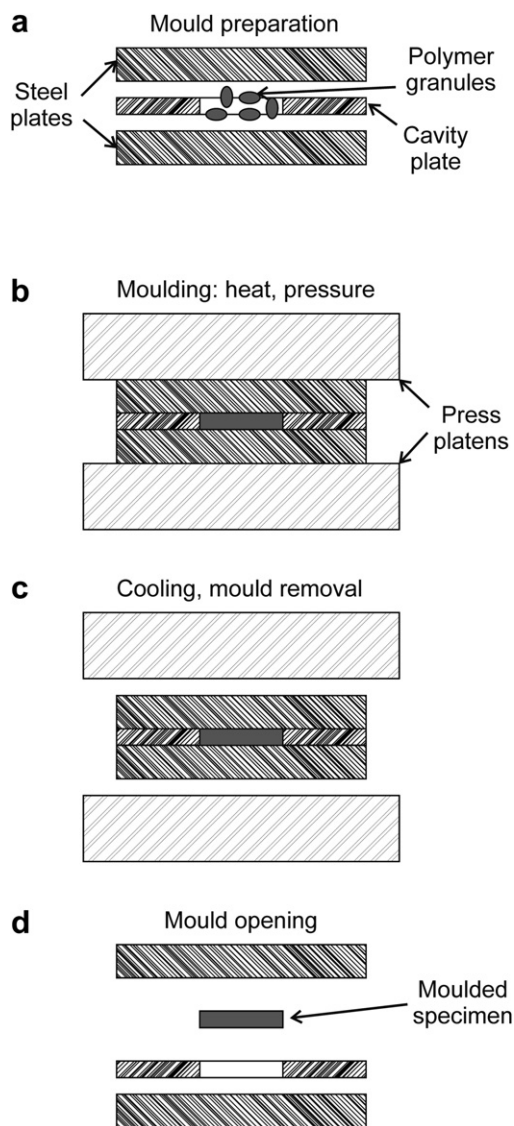


Fig. 1. The four stages in compression-moulding using a picture-frame arrangement: (a) the mould is prepared by loading an appropriate volume of polymer granules into a cavity; (b) the press is heated and pressure is applied to the mould; (c) the mould is cooled and the pressure is released; (d) the mould is opened and the moulded specimen is removed from the cavity.

moulds do not specifically exert pressure on the moulded polymer – they are designed so as to allow excess polymer (known as flash) to squeeze out of the mould cavity during moulding. Most of the applied compressive load is taken up by the metal picture-frame. Flash moulds are generally simpler to construct, as they can be manufactured out of sheet metal of appropriate thickness. The thickness of the moulded part is then equal to that of the metal picture-frame, unless substantial shrinkage occurs.

Positive moulds exert direct pressure on the polymer being moulded. The thickness of the moulded part depends on the precise volume of polymer in the cavity at the moulding temperature and pressure. Positive moulds require machining of cavities in solid metal parts, and are generally

more complex to construct. The removal of moulded specimens is challenging unless sufficient shrinkage occurs to draw the specimen away from the mould upon cooling, or the mould can be easily dismantled. Fig. 2 illustrates the basic design of flash and positive moulds.

2.3. Thermal transitions and shrinkage

Compression moulding, like most polymer processing operations, relies on achieving controlled and repeatable thermal transitions of the polymer being moulded, from the solid state to the molten state, and back to the solid state. There are fundamental differences in the way that a solid state is achieved that depend on whether the polymer being moulded is able to crystallise or not.

For amorphous or non-crystallising polymers, the moulding conditions need to be such that, during the heating stage, the temperature of the mould T_{mould} , and hence of the polymer, is sufficiently above the glass transition T_g that the polymer melt is able to flow and diffuse appropriately. One needs to ensure that granules or powders are able to melt and fuse at their interfaces, and that enough diffusion occurs for the resulting entanglement network to be considered uniform and isotropic. As a rule of thumb, at a given moulding temperature, a moulding time greater than three times the reptation time is considered appropriate for this purpose. For semicrystalline polymers, the moulding conditions during the heating stage need to ensure that any crystals present in the granules or powders prior to moulding are fully melted – this means that the mould temperature must exceed the melting temperature of the crystals T_m .

In the cooling stage, amorphous polymers need to be cooled to a temperature T_{open} sufficiently below the glass transition that they are rigid enough to handle. In order to ensure repeatability, this temperature is typically such that any effects of physical ageing are small. Semicrystalline polymers need either to be cooled below the glass transition of the amorphous fraction (if the degree of crystallinity is small) or, more typically, at an appropriate temperature rate and to a temperature such that sufficient crystal

domains have nucleated and grown so as to render the specimens rigid enough to handle. This means cooling to a temperature below the peak crystallisation temperature of the crystals T_m . In order to ensure repeatable specimens, moulds are typically cooled to a temperature at which further crystallisation occurs at a negligible rate. A representation of the changes in specific volume v that take place during the moulding process for both amorphous and semicrystalline polymers is given in Fig. 3.

The crossing of the thermal transitions is accompanied by volumetric changes in the polymer. On cooling, the shrinkage of amorphous polymers is determined first by the thermal expansion coefficient of the equilibrium liquid polymer melt (above the glass transition), and then by that of the out-of-equilibrium glass. The shrinkage of semicrystalline polymers is also influenced by the degree of crystallinity and by the change in density of the crystalline domains relative to their amorphous counterparts.

Shrinkage is a concern that needs to be addressed when compression-moulding. In positive moulds, the linear shrinkage can be countered by the application of an appropriate moulding pressure when the polymer is in the melt state, in a manner similar to the application of pressure during injection-moulding. The same cannot be easily performed with flash moulds, and these moulds are less suited to the production of polymer specimens where large degrees of shrinkage are expected. Although some shrinkage can help to release moulded specimens from the moulds, if excessive shrinkage occurs, cavitation of the polymer will occur.

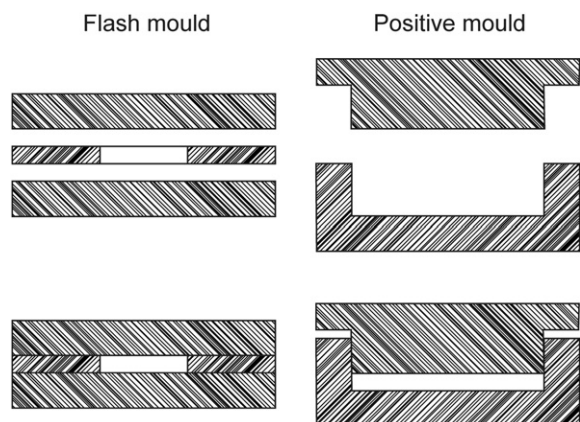


Fig. 2. Side view of the basic categories of moulds for compression moulding: the flash mould (also known as a picture-frame mould) and the positive mould.

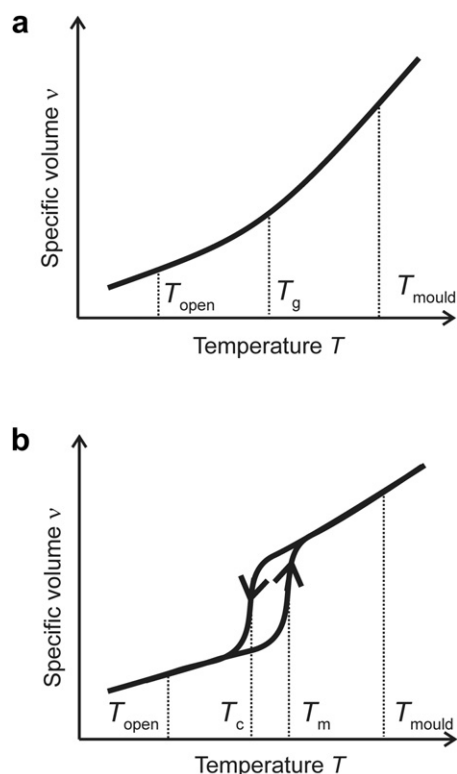


Fig. 3. Specific volume as a function of temperature for (a) a typical amorphous polymer, and (b) a typical semicrystalline polymer.

Generally, the shrinkage that occurs during the cooling of the mould is quantitatively smaller with amorphous polymers than with semicrystalline polymers. This makes amorphous polymers more suited to moulding with flash moulds, and semicrystalline polymers more suited to the use of positive moulds.

2.4. Specimen shapes

In principle, compression moulding can be used for the production of any required geometry of polymer specimens, but in practice the shapes that are achievable are dictated by two constraints: the complexity in the machining of the metal mould surfaces, and the difficulty in limiting damage to the moulded specimens when removing them from the moulds. These constraints pose limitations on the possible shapes achievable through the use of positive moulds. Although mould releases are commonly used [3], there may be occasions where contamination precludes their use. When dealing particularly with brittle polymers, positive moulds are limited to simple discs and blocks, unless substantial dismantling of the mould can be performed. Flash moulds can be more easily manufactured to form a wider variety of polymer shapes, and the limiting factor tends to be the ease with which the polymer specimens can be removed from the picture frame mould without damage – and without the use of a mould release agent if the test protocol demands it. In general, the greater the surface area of polymer in contact with the mould, the greater the challenge of removing the specimen.

3. Designs for near net-shape moulding

In this section we outline a series of designs for near net-shape compression moulding of polymer specimens. The designs are all based on the principle of flash moulds and consist of series of interlocking metal plates that form the picture-frame part of the flash mould. A set of two solid metal plates are used above and below the interlocking plates to contain the polymer and to mould the upper and lower surfaces of the moulded specimens. If allowed by the test protocol, a release spray, such as RS dry PTFE, is recommended to be used in order to aid the removal of the polymer specimens from the moulds. Alternatively, a release sheet can be used on the larger surfaces, above and below the picture-frame. The basic principle behind the novel moulds is that they allow the specimens to be separated from the moulds in stages, rather than in a single operation, thus reducing the force required for the separation of specimen and mould at any one time.

3.1. Basic single specimen mould

The basic design of a picture frame that enables the moulding of a single prismatic bar of polymer consists of two interlocking metal plates, a C-shaped part and a matching bar. When locked together, they produced a cavity of rectangular shape. After moulding, the two plates can be easily separated by pressing the bar down

relative to the C-shaped part. The basic design is illustrated in Fig. 4. The thickness of the moulded specimen is determined by the thickness of the picture-frame metal parts. For clarity, fillet radii are not shown in the diagrams that follow.

3.2. Single specimen variable width mould

A variation on the initial design consists of sets of interlocking C-shaped and H-shaped parts. Each H-shaped part can be made asymmetric, thus allowing for the production of specimens of two different widths with a single mould depending on the orientation of the H-shaped part. Additionally, several H-shaped parts can be manufactured to match a single C-shaped part, allowing multiple specimen widths to be achieved. The single specimen mould design with variable specimen width is shown in Fig. 5.

3.3. Multiple specimen moulds

Although the preliminary designs are effective in enabling near net-shape production of polymer specimens, they are inefficient in terms of specimen production rate, as they enable the manufacture of a single specimen per moulding. Where multiple specimens are required, an improved design is proposed that consists of an appropriately shaped outer picture frame and a series of rectangular metal inserts accurately located laterally within the outer frame through shaped slots, as shown in Fig. 6. The specimens produced in the cavities present between the metal inserts are rectangular in shape, and their thickness is again determined by the thickness of the metal parts. The illustrated design has 10 inserts, and 11 cavities.

3.4. Moulds for more complex specimen shapes

The designs illustrated so far are suitable only for the production of parallelepipedic bars. Although such prismatic bars are applicable to a wide range of test protocols, there are several other protocols that demand the production of more complex shapes. For example, the international

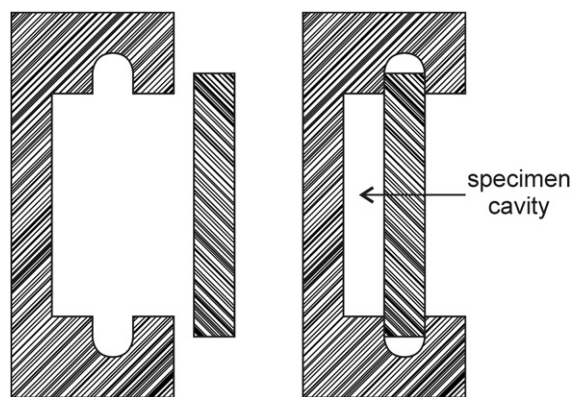


Fig. 4. Top view of the basic design of a picture-frame mould that allows for easier disassembly, consisting of two interlocking metal plates.

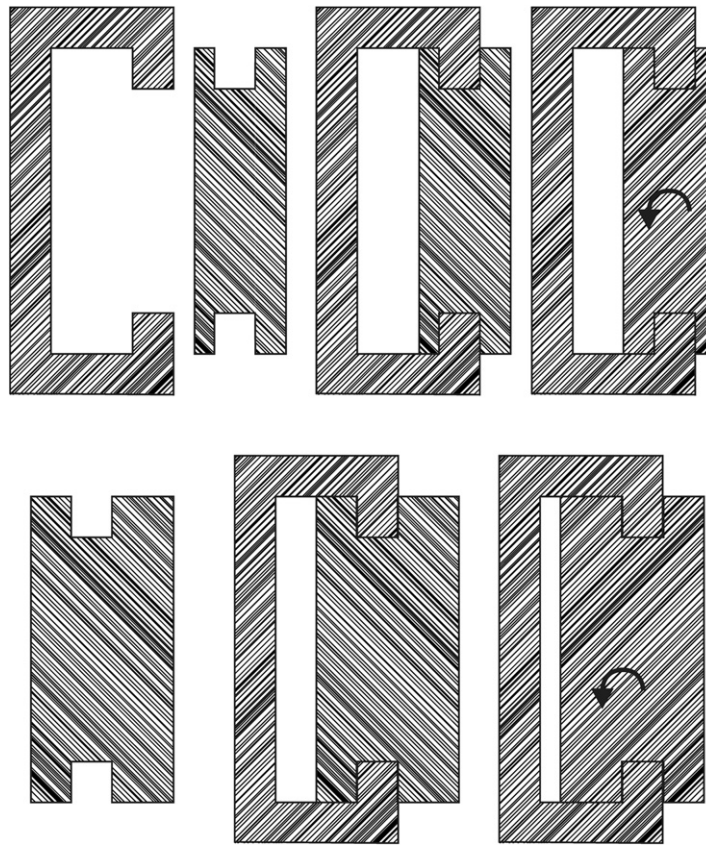


Fig. 5. Top view of a single specimen mould comprising a C-shaped part and two possible interlocking H-shaped parts. Each H-shaped part can be rotated by 180° to allow a second width of specimen to be moulded with the same set of mould parts.

standard for tensile testing of polymers recommends the use of dumbbell shaped specimens with a parallel-sided centre section and wider ends, primarily in order to avoid stress concentrations and failure at the grips [4,5]. It is possible to

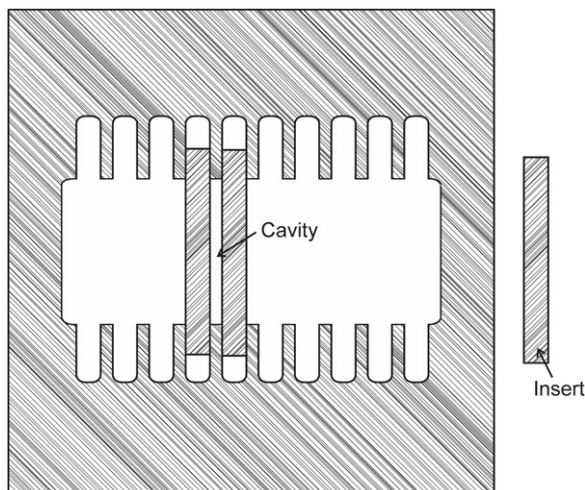


Fig. 6. Top view of a multiple specimen mould comprising an outer picture-frame in which interlocking inserts are located, leaving multiple cavities for specimen production.

adapt the design of the multiple specimen picture frame mould to cater for the production of dumbbell shaped specimens. In order to achieve this, two changes need to be implemented. The first is the introduction of protruding wings of the correct width and with correctly rounded inside and outside corners on both sides of the rectangular inserts fitting the outer picture-frame mould. The second is a re-design of the regions of the picture-frame where the inserts are located in order to ensure accurate positioning of these inserts along their long axis. This modification is illustrated diagrammatically for a single specimen cavity and two inserts in Fig. 7.

Through similar modifications to the basic design, it is possible to design moulds suitable for the manufacture of other shapes, such as notched and tapered specimens.

4. Discussion

4.1. Mould and specimen manufacture

Several of the aforementioned designs of mould have been commissioned for manufacture from stainless steel in our workshops. Single specimen moulds with variable widths, designed to produce rectangular bars 100 mm in length and either 6 mm or 8 mm in width, manufactured in two different thicknesses, 0.5 mm and 1 mm thick, have

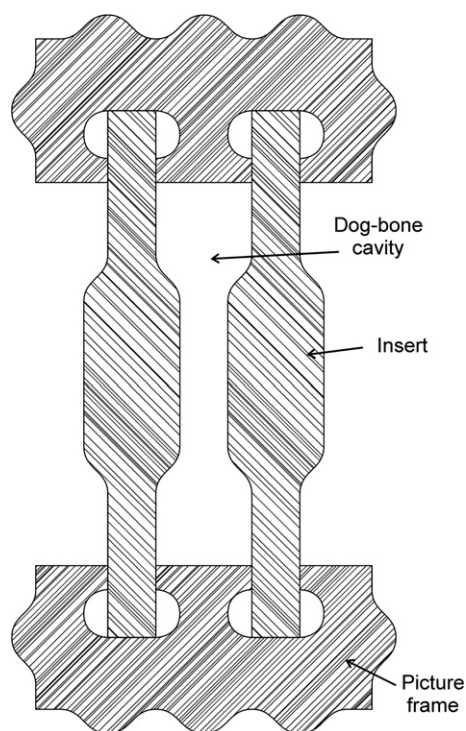


Fig. 7. Top view of a part of a multiple specimen mould in which especially shaped interlocking inserts are located, leaving dumbbell shaped cavities for specimen production.

been produced. The 1 mm versions are illustrated in Fig. 8. In order to ease manufacture, as well as accurate positioning, a number of fillet radii have been introduced into the design, and the outside corners of the C-shaped part that mate with the H-shaped part have been removed. Specimens produced with these moulds have two rounded corners, as a consequence of the manufacture of the moulds, as well as to ease specimen removal. If these are undesired, the multiple specimen moulds may be used

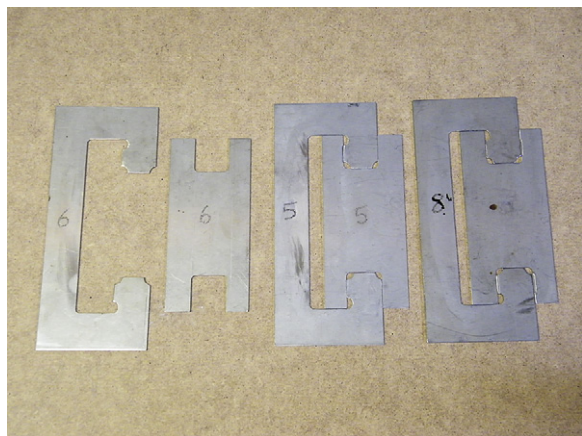


Fig. 8. C-shaped and H-shaped interlocking parts manufactured from stainless steel, for the moulding of rectangular bars 100 mm in length, and either 6 mm or 8 mm in width.

instead. These moulds were employed for the successful manufacture of several grades of polystyrene.

Two versions of the multiple specimen moulds, designed to produce 11 rectangular bars either 60 mm by 5 mm by 2 mm or 100 mm by 6 mm by 1 mm, have been manufactured, and are illustrated in Fig. 9, together with selected specimens manufactured from different grades of polystyrene using these moulds. The specimens from the ends of the multiple specimen moulds have two rounded corners, similar to those produced using the single specimen moulds, in order to ease removal, although these cavities can be left empty if this is undesirable.

4.2. Removal of specimens

In order to achieve void-free specimens, between 15% and 20% excess polymer is typically loaded into the cavities. This inevitably results in small amounts of flash polymer. The removal of the specimens after moulding is aided by gently cutting through the flash around the edges of the specimens with a sharp blade.

The single specimen moulds are most easily separated by rotating either the bar or the H-shaped part relative to the C-shaped part. The specimen typically remains attached to the C-shaped part, and can be removed by gently pressing on the free edge. When using the multiple specimen moulds, removal of the specimens is achieved by pressing the inserts away from the picture-frame. The specimens typically remain attached to the rectangular inserts, but are easily pried away from the individual inserts once these have been removed from the frame.

The sides of the moulded bars require smoothing after the removal process, with progressively finer grades of abrasive paper, in order to achieve flat surfaces. The ends of the bars can also be smoothed in this way if required by the test protocol. The success rate in producing void-free and undamaged specimens using these moulds is very high, typically in excess of 90%. The waste polymer generated is thus limited to 20–30%, and even less if the waste polymer can be re-moulded. This technique is, therefore, highly suitable for the moulding of limited quantities of polymer, or where the cost of the polymer is high.

4.3. Applicability of the technique

The moulds illustrated above were employed for the successful production of specimens from several grades of polystyrene, poly(methyl methacrylate), polycarbonate (including polycarbonate nanocomposites), poly(lactic acid) (with approximately 2% crystallinity after moulding) and polyurethane elastomers. Rectangular specimens produced using these techniques have been successfully employed in a range of studies, including 3-point bending creep and crazing experiments [6], dynamic mechanical analyses in 3-point bending and in cantilever modes [6], and extensive tensile testing [7]. Dumbbell specimens have not yet been manufactured using this technique. Using moulding protocols as described above, all transparent specimens produced were verifiably optically isotropic [8], and visible damage such as crazing and cracking was eliminated even in relatively brittle grades of polystyrene

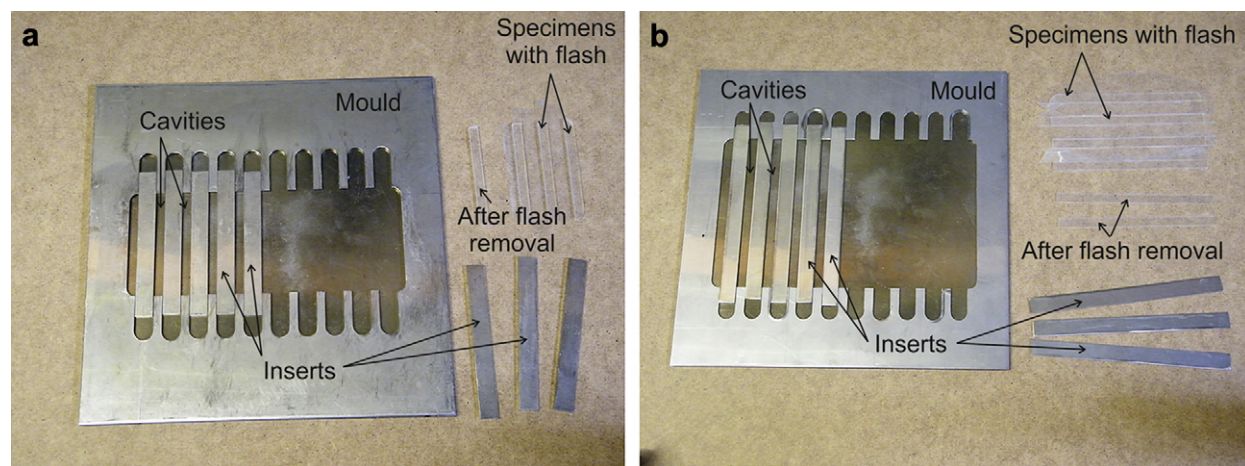


Fig. 9. Multiple specimen moulds designed to produce 11 rectangular bars of dimensions (a) 60 mm by 5 mm by 2 mm, or (b) 100 mm by 6 mm by 1 mm, shown with selected specimens before and after flash removal. The specimens illustrated were moulded from different grades of polystyrene.

[9]. No post-moulding machining (other than minimal clean-up of the sides using abrasive papers) was required and, therefore, no subsequent annealing was necessary.

5. Conclusions

This study has presented a range of designs for the near net-shape manufacture of polymeric specimens using novel variations on the traditional flash mould design. The designs consist of interlocking metal parts that allow for precise cavities to be formed within the mould, and that are easily dismantled after moulding. Mould designs suitable for the production of both single and multiple rectangular prismatic specimens have been described. Polymeric specimens manufactured using the moulds described have been successfully produced and employed for a wide range of experimental characterisation techniques. Mould designs suitable for the production of dumbbell specimens and for multipurpose test specimens have also been presented. Other geometries are also possible with minor modifications. The proposed designs are of particular value where the polymer is brittle, is available in limited quantities, is very expensive, where post-moulding machining and annealing is to be kept at a minimum, and where damage to the specimens during removal from the moulds is to be reduced.

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